Syllabus, MSNE/PHYS 435/535, Spring 2015 rev. 1/7/2015 PJL

Crystallography and Diffraction – Study of crystals by x-ray, electron and neutron diffraction. Includes basic diffraction theory as well as methods for characterizing the structure, composition and stresses in crystalline materials.

This course is required for all MSNE undergrads and most MSNE grad students. It may also be appropriate for students in CHEM, CENG, PHYS, ELEC & GEOL. For biology students there is BIOC 535.

- **Instructor:** Peter Loos **on campus:** ME 215 ploos@rice.edu **Office hours:** after class and by appointment
- **Class Meets:** MWF 10:00-10:50am in MEB 128
- **Prerequisites: None.** However MSCI 301 is recommended. A good understanding of geometry and trigonometry is essential. Differentiation, integration and complex arithmetic are also used.
- **Text: *Elements of X-ray Diffraction***, 3rd ed. by Cullity & Stock (2001). Available from abebooks.com, bn.com, amazon.com, www.campusbooks4less.com, directtextbook.com, etc. but not Rice Campus Store. Cost of the international edition is less than $20. 2nd edition can also be used along with a cross-reference list. The various 3rd editions have more errors than 2nd edition. See errata list. **The 1956 1st edition is *NOT* suitable.**
- **Grading: 435 class:** 45% homework, 45% tests, 10% quizzes. **535 class:** 35% homework, 35% tests, 20% term paper, 10% quizzes. There will be approximately two tests, three quizzes and eleven homework assignments. Points may be deducted for assignments turned in late.
- **Absence Policy:** Missed quizzes and tests will have to be made up right away.
- **Honor Code:** Tests and quizzes must be entirely your own work. The Rice Honor Pledge must be included on every one. Homework may be ***discussed*** with others, but you must NOT look at or copy from anyone else's written homework solutions. Students must write their own papers per the Rice Honor Code.
- **Software:** CaRIne software is used to display crystal structure models and the corresponding reciprocal lattices, powder diffraction patterns, etc. PowderCell has many of the same features and is available free. See item below under Resources on the Web. Spreadsheets will be used extensively.
- **Lab:** MSNE 537 is required for the BS MSNE and open to all 435/535 students. Meeting times TBD. **Location:** Space Science basement room B-06, the SEA X-ray Scattering Facility.
- **Students' Evaluations:** see esther.rice.edu . The last time Instructor taught this class was Fall 2011.

Lectures & Topics

1 Introduction.
1 Production, discovery & applications of cathode rays & x-rays.
1 Properties of x-rays. Emission, absorption, detection & safety precautions.
2 Crystal lattices and their symmetry. Point groups and space groups.
2 The reciprocal lattice and stereographic projection.
2 Diffraction, constructive & destructive interference, derivation of Bragg's Law. Experimental methods for single & polycrystalline samples, effect of crystallite size (thickness) per Scherrer's eqn.
2 Scattering of x-rays by electrons, atoms and unit cells.
3 Structure factor for various crystals, other factors affecting intensity of diffracted beams, application to powder diffraction.
1 Use of CaRIne or PowderCell software. Use of intensity ratios to determine volume fractions of phases present.
3 Indexing of diffraction patterns and determination of crystal structure. Precise determination of lattice parameters.
2 Determination of phase diagrams. Ordered and disordered alloys.
1 Measurement of micro-strain and residual macro-stress. Separation of line broadening due to micro-strain and crystallite size.
2 Comparison of x-ray, electron & neutron diffraction methods; scattering factors for electrons; Ewald construction in reciprocal space.
2 Selected area diffraction (SAD) on the transmission electron microscope (TEM), indexing of SAD patterns.
2 Small angle x-ray scattering (SAXS)
1 Quasicrystals, discovered in 1984

Goals for this course:

To address ABET Student Outcomes and Program Criteria, students will have

(a) an ability to apply knowledge of mathematics, science, and engineering
(e) an ability to identify, formulate, and solve engineering problems

(g) an ability to communicate effectively (MSNE 535 only)
(k) an ability to use the techniques, skills, and modern engineering tools necessary for engineering practice

and the course will

- prepare graduates to apply advanced science … and engineering principles to materials systems …
- prepare graduates to utilize experimental, statistical, & computational methods …

More specifically:

(1) Students will become familiar with the crystal structure of most metals and ceramics, how that structure is determined experimentally, and how it is described in the literature.
(2) Students will become familiar with how such structure information is used to determine strains, stresses, composition, orientation, density, and size of features in the crystalline solid.
(3) Students will be able to assess the technical literature on this subject.

Optional Supplemental Reading

X-Ray Diffraction by B.E. Warren, Dover 1990 (Addison-Wesley, 1969) $17 ...rigorous graduate-level text, detailed mathematical theory, includes thermal vibration effects, order-disorder phenomena, crystal imperfections, structure of amorphous materials, and diffraction of x-rays in perfect crystals. If requested, may be placed on reserve at Fondren library.

Fundamentals of Crystallography by Giacovazzo, Monaco, Viterbo, Giacovazzo & Scordari, Oxford U. Press, 1992 $34 ...advanced graduate level text from IUCr, includes crystal symmetry, computational methods, physics of diffraction, instrumentation & methods, ionic and molecular crystals, protein crystallography, etc.

Introduction to Diffraction in Materials Science and Engineering by A. D. Krawitz. A well-written book, intended as a one-semester introduction, on a level comparable to our text, but with more emphasis on neutron diffraction and little concerning electron diffraction. If requested, may be placed on reserve at Fondren library.

Solid State Physics by Ashcroft & Mermin (Holt Rinehart Winston, 1976). See chapters 4,5,6,7,19,20 and Appendices F & N.

Metals Handbook 9th ed. by ASM Handbook Committee, Volume 10, ***Materials Characterization*** See: "X-Ray Spectrometry" p. 82, "X-Ray Powder Diffraction" p. 333, "Single Crystal X-Ray Diffraction" p. 344, "Neutron Diffraction" p. 420, "Analytical Transmission Electron Microscopy" p. 429

Metals Handbook 8th ed. by ASM Handbook Committee, Volume 8, ***Metallography, Structures and Phase Diagrams***. See: "Crystal Structure" p. 233-250, "Ordered Structures" p. 205-206, "Textured Structures" p. 229-232. ... The Metals Handbook is a great resource for all practicing crystallographers, materials scientists, metallurgists, and ceramicists.

The Basics of Crystallography and Diffraction by C. Hammond, Oxford. Univ. Press and Int. Union of Crystallography, 1997 $72 (paperback) ...a good introductory undergraduate-level text which avoids much of the intricate mathematical theory.

Introduction to Crystallography by Donald E. Sands. Another nice $12 paperback from Dover. Includes two chapters on the theory and methods of x-ray diffraction.

Quasicrystals: A Primer 2nd Ed. by C. Janot, Oxford Univ. Press. $55

The Annual Book of ASTM Standards. 77 volumes containing about 12,000 "standards" documents relating to the production, testing, and performance of countless materials of commercial importance. For details see http://www.astm.org/ or Annual Book of Standards or Standards Search. Fondren has the entire set on microfilm and your instructor has nine volumes in paperback. About 20 or so ASTM standards involve XRD, everything from instrument alignment to measurement of stress, particle size, composition and orientation. See list at H&M Analytical.

Crystallography Resources On the Web

- Crystallography of proteins and other large molecules at Rice. See BIOC 535 description at the Registrar's web pages.
- Discovery of the Electron, historical developments from the AIP: www.aip.org/history/electron/
- Int. Union of Crystallography: Links to everything crystallographic. www.iucr.org. See especially Educational Resources at www.iucr.org/cww-top/edu.index.html for example, www.iucr.org/iucr-top/comm/cteach/pamphlets/16/index.html, The Study of Metals and Alloys by X-ray Powder Diffraction Methods.
- XRD on Mars: www.iucr.org/news/research-news/the-first-x-ray-diffraction-measurements-on-mars and http://journals.iucr.org/m/issues/2014/06/00/yu5005/index.html and https://www.youtube.com/watch?v=20zeFVQ87ro starting at 41:10.

- American Crystallographic Association – see www.amercrystalassn.org
- Crystal lattice structures from the Naval Research Lab. These were taken offline a few years ago and have to be accessed through the Wayback Machine at archive.org/web/: See http://cst-www.nrl.navy.mil/lattice/ also many useful links at: http://cst-www.nrl.navy.mil/lattice/others.html. The versions before March 2010 seem to be OK.
- Crystallographic Space Group Diagrams & Tables http://img.chem.ucl.ac.uk/sgp/mainmenu.htm from University College London
- XRayView, a virtual X-Ray crystallography laboratory from George Phillips: www.bioc.rice.edu/Bioch/Phillips/Papers/XRayView/
- LawrenceBerkeley: X-ray interactions with matter, data & calculations henke.lbl.gov/optical_constants/ including x-ray reflectivity modeling software.
- Tutorial on protein crystallography www.ruppweb.org/Xray/101index.html from B. Rupp at QED Life Sciences
- PowderCell software from the German Fed. Inst. for Materials Research & Testing. Download for free. www.bam.de/de/service/publikationen/powder_cell.htm
- International Centre for Diffraction Data - purveyors of the Powder Diffraction File (PDF) www.icdd.com
- Crystallography Open Database: www.crystallography.net searchable and free but limited. Results are given in the CIF (crystallographic information file) ASCII text format. See www.iucr.org/resources/cif for details.
- Collaborative Computational Project Number 14: www.ccp14.ac.uk Lots of helpful tutorials and free software for single crystal and powder diffraction.
- **Mineral structural information:** MINCRYST, searchable by name, composition & other parameters: database.iem.ac.ru/mincryst/ and webmineral.com which is full of useful mineralogy & crystallography information and also the American Mineralogist Crystal Structure Database http://rruff.geo.arizona.edu/AMS/amcsd.php
- **Quasicrystals:** Introduction by S. Weber: jcrystal.com/steffenweber/qc.html and also by Ron Lifshitz at TAU: www.tau.ac.il/~ronlif/quasicrystals.html .
- **Commercial XRD labs:** Lambda Research, founded by Cullity www.lambdatechs.com. See especially publications www.lambdatechs.com/publications/publications.html. Rigaku, who sell XRD instruments and also provide XRD measurement services www.rigaku.com/apps/. H&M Analytical Services www.h-and-m-analytical.com. All three labs provide plenty of application notes, papers and other technical literature for free download.

NOTICE

Any student with a disability requiring accommodations in this course is encouraged to contact me after class or during office hours. Additionally, students will need to contact Disability Support Services in the Ley Student Center.